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Flow injection solid phase extraction with Chromosorb 102: determination of lead in soil and waters by flame atomic absorption spectrometry ☆

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Abstract

A flow-injection, solid-phase extraction, preconcentration method was developed for the determination of lead in soil and water samples by flame atomic absorption spectrometry. Lead, in the form of the diethyldithiocarbamate complex, was retained on a miniature column of Chromosorb 102 from buffered sample solutions. Several parameters governing the efficiency and throughput of the method were evaluated including pH, eluent composition, volume and flow rate. The complex retained from pH 9.0 (ammonium acetate) solutions was eluted with 176 μl of ethanol, at 4.2 ml min^{-1} into the nebulizer–burner system of the spectrometer. The detection limit was 2 μg l^{-1} for preconcentration at 2.2 ml min^{-1} for 120 s. The relative standard deviation (R.S.D.) was less than 4% for concentrations down to 400 μg l^{-1}. The procedure was validated by the analysis of NIST standard reference material 2711(Montana Soil) which contains lead at a concentration of 1162 μg g^{-1}. Recoveries of spike additions (100–400 μg l^{-1}) to artificial sea water and tap water were quantitative.

Keywords: Flow injection; Solid phase extraction; Pb; Soil; Flame atomic absorption spectrometry

1. Introduction

There is an on-going need to determine lead because of its extensive distribution and high toxicity. Flame atomic absorption spectrometry (FAAS) is, in principle, a suitable technique as it is rapid, simple and inexpensive; however, lead concentrations in matrices such as biological materials, soil, natural water, environmental, and food samples are such that, typically, the concentrations in sample extracts or digests are below the detection limit of FAAS. In addition, high concentrations of matrix components (including the acids used for sample dissolution or digestion) may cause inaccuracies in a method which is

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based on external calibration with dilute aqueous standards. Therefore, a preconcentration and/or separation is needed to improve the detection limit and selectivity for the determination of lead by FAAS. Flow injection (FI) techniques for preconcentration and separation coupled with FAAS have been used for this purpose [1,2], and several solid phase extraction (SPE) procedures have been developed for the determination of lead [3–12]. The most extensively used extractant is C-18 silica [8–12], though various other materials, including activated carbon [13], alumina [14], cellulose [15], and Amberlite XAD-8 have been used [16].

It would appear that the most successful general approach is to use a hydrophobic solid phase extractant for the retention of a non-polar derivative of the target analyte. A class of hydrophobic materials that, to our knowledge, has not been investigated for use in FI-SPE procedures is the various non-polar gas chromatography (GC) stationary phases. Chromosorb 102, (a styrene–divinyl-benzene copolymer) has been extensively used in gas chromatography for the separation of organic compounds such as hydrocarbons, alcohols, pesticides and aromatics in a variety of samples [17–19].

To demonstrate the feasibility of this material as a solid phase extractant for use in FI-AAS, we have used it in a procedure for the determination of lead by retention from alkaline solution as the diethyldithiocarbamate complex, followed by elution with a discrete volume of ethanol. The procedure was applied to the analysis of tap water, artificial sea water, and a certified reference soil. In the development of the procedure, a number of parameters relevant to the retention and elution of the analyte were investigated, but no attempt was made to develop a fully automated procedure.

2. Experimental

2.1. Apparatus

A Perkin-Elmer (Norwalk, CT, USA) Model 1100 B atomic absorption spectrometer with deuterium background correction, equipped with a 10-cm air–acetylene burner, was used. An intensitron (Perkin-Elmer) lead hollow cathode lamp, operated at 7 mA, was used at 283.3 nm with a 0.7-nm spectral bandpass. Data were recorded by an Epson Model LQ-850 printer. An air–acetylene flame was employed with gas flow rates of 6.0 and 2.5 l min^{-1} for air and acetylene, respectively. The nebulizer uptake rate and position of the capillary in the venturi throat were adjusted to obtain the maximum absorbance for conventional sample introduction (approx. 6.0 ml min^{-1}).

A Perkin-Elmer FIAS-200 unit with a six-port rotary flow injection valve was used as the sample introduction system. The schematic diagram of the FI manifold for preconcentration and matrix

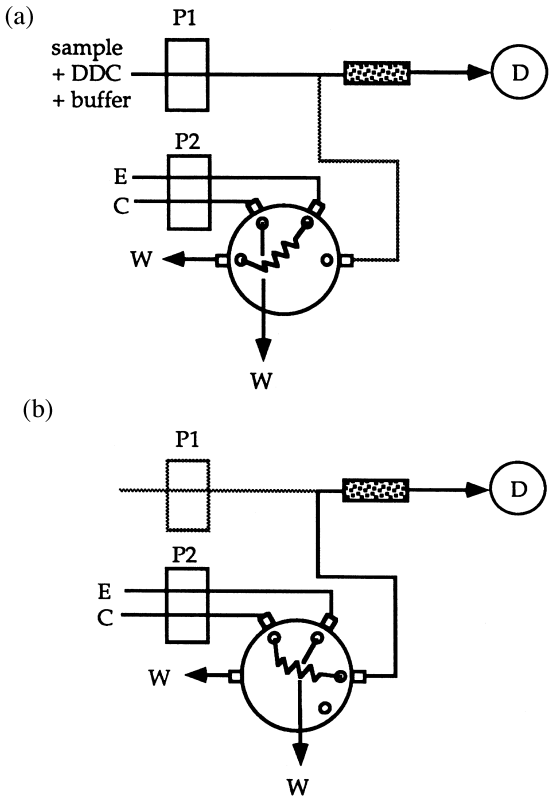

Fig. 1. Schematic diagram of the flow injection manifold: P_1, sample pump; P_2, eluent and carrier pump; D, detector; C, carrier; E, eluent; and W, waste. (a) Sample loading position; after pump 1 is turned off, pump 2 runs for 10 s to fill the injection loop. (b) Elute position; the valve is turned to deliver a discrete volume of the eluent.

separation is shown in Fig. 1, and the FIAS program is given in Table 1. Tygon pump tubes (1.52 mm i.d.) were used for sample, ethanol and carrier solutions. All connecting tubings were made of 0.8 mm i.d. PTFE.

For the microwave digestion of the soil samples a CEM (Indian Trail, NC, USA) Model MSD-81D oven, providing 630 ± 70 W output power at the 100% power setting, was used. The 12-vessel turntable drive system rotates the samples 360° within the microwave field at 6 rev. min^{-1}. The vessel body and the cap are made of polyetherimide (Ultem), and the liner, cover and the rupture membrane are made of Teflon®-perfluoralkoxy (PFA). The vent screw is constructed of PTFE. The volume of the liner is 100 ml.

2.2. Reagents and samples

All reagents used were of analytical reagent grade. High-purity deionized water (18 MΩ cm) was used throughout the experiments. Diethyldithiocarbamic acid diethyl ammonium salt (Aldrich) was used for daily preparation of 0.5% m/v NH_4DDC solution by dissolving 0.05 g of solid NH_4DDC in ammonium acetate buffer solution (0.06 M NH_3 + 0.03 M CH_3COOH, pH 9). The DDC solution was passed through a column packed with Chromosorb 102 resin to eliminate possible contamination prior to the addition into standard and sample solutions. Standard solutions of lead were prepared daily in the concentration range of 0.05–0.6 μg ml^{-1} by appropriate dilution of a 1000-mg l^{-1} stock solution (Aldrich). Ethanol was used as the eluent.

Chromosorb 102 resin, a porous styrene–divinyl-benzene copolymer, (Phase Separations Inc., Norwalk, CT, USA) having a surface area of 300–400 m^2 g^{-1} and a particle size of 80–100 mesh was used as the solid phase extractant. This hydrophobic resin can be used in a pH range from 0 to 14 [20]. Artificial sea water (250 ml) was prepared by dissolving 0.3675 g of $CaCl_2 \cdot 2H_2O$, 3.275 g of $MgSO_4 \cdot 7H_2O$ and 6.975 g of NaCl in deionized water [21]. The pH values of the test solutions were adjusted to pH 9.0 by dropwise addition of NH_3 and CH_3COOH solutions before use.

2.3. Method development

The column, a glass tube of 45 mm long and 2.3 mm i.d., was dry-packed with 110 mg of Chromosorb 102 resin. A small amount of glass wool was placed at both ends of the column to prevent loss of the resin beads during sample loading. The

Table 1
FIAS-200 program

Step no.	Time (s)	Pump 1 (rev. min^{-1})	Pump 2 (rev. min^{-1})	Valve position	Remarks
1	Variable	50	Off	Fill	Preconcentration — sample pumped through column for a maximum of 99 s
2	Variable	50	Off	Fill	Second stage needed for preconcentration time greater than 99 s
3	10	Off	100	Fill	Injection loop filled with ethanol, carrier flushes unretained components from column
4	15	Off	100	Elute	Valve switches to elute position, slug of ethanol flushed through column

resin bed was approximately 40 mm long. For the conditioning of the column, ammonium acetate buffer solution (pH 9.0) which contained 0.5% m/v DDC, was passed through the column which was then washed with ethanol and water several times. After each elution, the column was ready for re-use without further pretreatment. In addition, the performance of the column was evaluated daily by preconcentrating a 0.4-μg ml^{-1} Pb solution for 60 s.

The effect of pH on the retention of lead was examined over a pH range of 4–10. Lead standard solutions containing 0.4 μg ml^{-1} Pb were loaded onto the column after complexing with DDC. The flow rate of the sample solution (0.4 μg ml^{-1} lead) was varied from 1.3 to 5.0 ml min^{-1} to investigate the effect on the retention of lead on the column. The volume of the eluent was varied over the range of 50–200 μl, and the eluent flow rate, for 176 μl of ethanol, was varied from 2.9 and 5.0 ml min^{-1}. For the elution of the Pb–DDC complex from the column, ethanol, ethanol + 2% v/v HCl mixture (1 + 1), 1% v/v HCl, and 4% HNO_3 were investigated. The flow rate of the carrier during these experiments was 2.2 ml min^{-1}. The volume of 0.5% m/v DDC solution added to a 25-ml sample or standard solution was varied from 0 to 1000 μl, while that of the buffer solution was varied from 0 to 25 ml.

For most studies, the figure of merit was maximum peak height; but, in the studies of the amount of reagents, the value of the blank signal was also taken into account.

2.4. Method validation and procedures

A NIST soil reference material (SRM 2711, Montana Soil) was used for method validation. The soil was dissolved by a closed-vessel, microwave-assisted, digestion procedure [22]. Approximately 100 mg of the soil sample was digested in a mixture of 5.0 ml of sub-boiled HNO_3 and 1.5 ml of HF at 240 W for 10 min [22]. At the end of the program, the vessels were removed and cooled to room temperature in a fume hood. After venting, the vessels were again tightly capped and irradiated for a further 10 min. This step was repeated twice. At the end of the third step, complete dissolution was achieved yielding colorless solutions. The contents in the vessels were then diluted to 50 ml with water in a polyethylene calibrated flask. For the determination of Pb, either 1.0 or 2.0 ml of the original 50-ml solution was first neutralized by dropwise addition of the required volume of ammonia solution, and then diluted to 50 ml following the addition of 10.0 ml of the buffer solution (pH 9) and 100 μl of 0.5% m/v DDC solution.

For recovery studies performed with artificial sea water, 8 ml of the buffer solution (pH 9) was first added to 40 ml of the sea water which was then spiked with either 5, 10 or 20 μl of Pb (1000 μg ml^{-1}). The solution was diluted to 50 ml with the buffer solution after adding 100 μl of 0.5% m/v DDC solution. The concentration of Pb in the analysis solutions was either 0.1, 0.2 or 0.4 μg ml^{-1}. Recovery studies were also performed with laboratory tap water. No further acid pretreatment was made for the tap water samples. All pH adjustments and additions were made as for the artificial sea water samples.

Sample and standard solutions were loaded onto the column with the FI manifold shown in Fig. 1. In the preconcentration step, the sample solution containing the lead–DDC complex in the buffer solution was pumped by Pump 1 at 2.2 ml min^{-1}. The effluent was directly introduced to the nebulizer of the spectrometer. During the last 10 s of the preconcentration step (Step 3), Pump 2 was turned on and the loop of the valve filled with ethanol while the carrier (water) was directed to waste. The flow rate for both ethanol and water was 4.2 ml min^{-1}. In the elution step, the valve was switched to the elute position, and the ethanol in the loop was flushed through the column by the water carrier. The Pb–DDC complex was eluted and introduced to the spectrometer. During elution, the carrier stream conditioned the column for the next preconcentration.

External calibration was performed with Pb standard solutions in the concentration range of 0, 0.05, 0.1, 0.2, 0.4 and 0.6 μg ml^{-1}. Peak height absorbance was measured and five replicate measurements were made for each standard and sample solution. The calibration data were then processed on a personal computer using Excel

(Version 5.0) spreadsheet to obtain the calibration curve and the equation of calibration. The mean peak height absorbance for each sample solution was then substituted into the calibration equation to calculate the concentration of lead. Peak height was used as it was found that, for the sharp peaks typically obtained, the signal-to-noise ratio was better than that of peak area measurements. With the data handling capability available it was not possible to employ gated integration, and thus the integration window for peak area was sub-optimal.

3. Results and discussion

3.1. Effect of chemical variables on preconcentration

The effect of the pH on the retention of Pb–DDC complex is shown in Fig. 2 for a 0.4-μg ml^{-1} Pb standard solution. The maximum signal was obtained in basic solutions, in the pH range from 8 to 10. Thus, the pH was adjusted to 9 for subsequent experiments. For the various eluents, the highest signal was achieved with ethanol. Lower signals and incomplete elution (as evidenced by significant peaks for replicate elutions) of the Pb–DDC complex was observed for all the other solutions including ethanol +2% HCl mixture (1 + 1), 1% v/v HCl, and 4% v/v HNO_3, which resulted in lower relative signals (approximately 45–60%). The volume of ethanol which gave the highest signal was 176 μl. For lower volumes, relative signals were lower, probably due to incomplete elution. For volumes greater than 176 μl, the signal decreased gradually because of the subsequent dilution of the analyte. No memory effects were observed for elution with 176 μl of ethanol. The column was ready for the next sample after passing deionized water for 10 s following the appearance of the Pb signal.

The effect of sample flow rate (0.4 μg ml^{-1} Pb) is shown in Fig. 3. It can be seen that retention of the Pb–DDC complex was independent of flow rate from 1.3 to 2.2 ml min^{-1}. Flow rates greater than 2.2 ml min^{-1} resulted in lower signals which were interpreted as incomplete retention of the

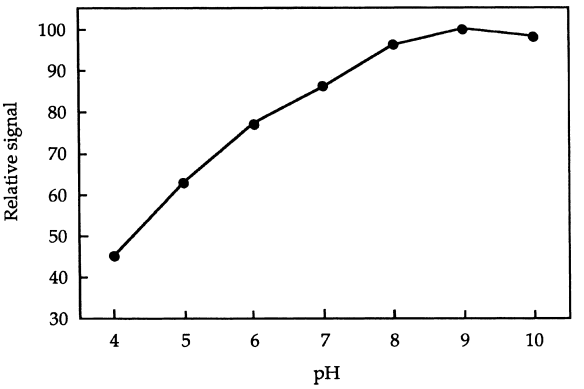

Fig. 2. Effect of pH on retention of Pb–DDC complex (0.4 μg ml^{-1} Pb solution).

Pb–DDC. Thus, the flow rate was kept at 2.2 ml min^{-1} during the experiments. The effect of the eluent (ethanol) flow rate on the signal is also shown in Fig. 3. Maximum signal was achieved for flow rates between 3.8 and 5.0 ml min^{-1}. For lower flow rates, the Pb signals were always lower. This was attributed to reduced nebulizer efficiency and dispersion of ethanol into the carrier stream, which could have resulted in the incomplete dissolution of the Pb–DDC complex. A carrier flow rate of 4.2 ml min^{-1} was used.

The results for the effect of the concentration of 0.5% m/v DDC solution added into the 25-ml sample solution (0.4 μg ml^{-1} Pb) are given in Table 2. The minimum volume needed to achieve the maximum signal was 50 μl, which yielded a

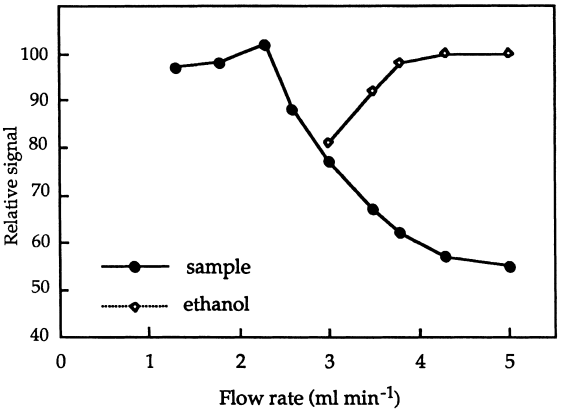

Fig. 3. Effect of sample and eluent flow rates.

Table 2
Effect of amount of DDC and buffer solutions on relative signal (25 ml sample volume solution; 0.4 μg ml^{-1} Pb)

0.5% m/v DDC solution		Buffer solution (pH 9.0)	
Volume (μl)	Relative signal (%)	Volume (ml)	Relative signal (%)
0	56	0.0	55
50	100	1.0	67
100	100	2.5	76
200	95	5.0	99
500	100	10.0	100
1000	98	15.0	95
		25.0	100

DDC concentration of 0.001% m/v in the 25-ml solution. No significant differences were observed in the Pb signals when the volume of DDC solution was increased up to 1.0 ml. Although Pb was retained on the column even in the absence of DDC, the relative signals were low (approx. 56%). This phenomenon was not investigated in further in this study; however, it has been examined as the basis of a possible multi-element preconcentration procedure [23]. The minimum volume of the ammonium acetate buffer solution to adjust the pH of the solution to 9 was 5 ml for a 25-ml sample solution (Table 2). Higher volumes were not used to avoid contamination from the buffer solution.

3.2. Analytical performance

The FI on-line solid phase extraction/preconcentration method developed allowed the determination of Pb in the concentration range of 0.05–0.6 μg ml^{-1} by FAAS for 120-s sample loading at 2.2 ml min^{-1}. The calibration equation was $A = 0.0045 + 0.0534$[Pb, μg ml^{-1}] ($r = 0.9973$). Without preconcentration, for 1.0–2.0 μg ml^{-1} Pb, the calibration equation was $A = 0.0015 + 0.0021$[Pb, μg ml^{-1}] ($r = 0.9968$). The experimental enhancement factor, calculated from the ratio of the slopes of the calibration equations, was 25.4. The theoretical preconcentration factor, calculated as the ratio of the sample volume (4.4 ml) to the eluent volume (0.176 ml), was 25.0. The experimental and theoretical preconcentration factors obtained for 60-, 90- and 120-s sample loading are summarized in Table 3. The agreement between the enhancement factors and the preconcentration factors is probably fortuitous as the transient signal for the FI procedure depends on the mass flux entering the flame as a function of time, which in turn is related to the kinetics of dissolution, the dispersion in the flow lines and the kinetics of the nebulizer and spray chamber response. In addition, the FI signal is generated from an ethanol solution whereas that for the conventional introduction is generated from an aqueous solution for which the nebulization efficiency is most likely different. It could be argued that, to a first approximation (equal nebulization efficiencies for water and ethanol, and rapid spectrometer response), the agreement demonstrates that for a 176-μl elution volume the response is pseudo-steady state with the eluted lead distributed uniformly throughout the ethanol slug. Although the column lifetime was not studied in detail, no significant changes were observed in the retention properties during the course of the study (preconcentration of lead at different concentrations at least 800 times). The difference in daily absorbance values for a 0.4-μg ml^{-1} Pb standard was not greater than 5%. The column was used as long as the same peak height absorbance was obtained (up to 20 days).

The precision expressed as the R.S.D. for nine replicate measurements of a 0.4-μg ml^{-1} Pb solution was 2.5% for a 60-s loading time. The signals for 60-, 70-, 90-, 120-, and 130-s loading times were directly proportional to the mass of lead

Table 3
Experimental and theoretical preconcentration factors for different sample loading times

Loading time (s)	Factor	
	Enhancement[a]	Preconcentration[b]
60	12.9	12.5
90	19.4	18.8
120	25.4	25.0

[a] Calculated as the ratio of the calibration slopes.
[b] Calculated as the ratio of the volumes.

introduced to the column. The limit of detection, based on the 120-s preconcentration of the ammonium acetate buffer blank, was 2.5 $\mu g\ l^{-1}$ (calculated as the concentration giving a net signal equal to three times the standard deviation of 10 replicate measurements of the blank signal).

3.3. Results for soil and water samples

The concentration found was $1147 \pm 46\ \mu g\ g^{-1}$ for six separate determinations of the lead in NIST Montana Soil, SRM 2711. The certified concentration is $1162 \pm 32\ \mu g\ g^{-1}$. The $\pm$ terms are the 95% confidence limits. Thus as the confidence interval for one value includes the mean of the other a *t*-test would indicate no significant difference between the values. The percent R.S.D. was 3.8% ($n = 6$). This result indicated that the developed method was not affected by the potential interferences from the major matrix elements, including Al (6.53%), Ca (2.88%), Fe (2.89%), Mg (1.05%), K (2.45%), and Na (1.14%).

The procedure was further validated by the determination of Pb in tap water and artificial sea water samples. The artificial sea water contained 400–11 000 $\mu g\ ml^{-1}$ Na, Ca and Mg as their chlorides or sulfates. The results are given in Table 4. Quantitative recoveries (94–104%) of various lead spikes were obtained, showing that the presence of high concentrations of alkaline and alkaline earth metals had no significant effect on the retention performance of Chromosorb 102 for the lead–DDC complex.

Table 4
Analysis of artificial sea water and tap water samples spiked with Pb

Sample	Added ($\mu g\ ml^{-1}$)	Found ($\mu g\ ml^{-1}$)	Recovery (%)
Artificial sea water	0	< D.L.	–
	0.100	0.098	98.4
	0.200	0.191	95.6
	0.400	0.409	102
Tap water	0	< D.L.	–
	0.100	0.104	103
	0.200	0.196	98.4
	0.300	0.283	94.3
	0.400	0.377	94.3

4. Conclusion

The studies indicate that Chromosorb is a robust and stable hydrophobic material capable of retaining lead–DDC from aqueous solution. The presence of high concentrations of Al, Ca, Fe, K, Mg and Na can be tolerated. Thus, the material has potential as a general-purpose, solid-phase extractant as it is likely that other metal DDC complexes (or other bulky, neutral, non-polar derivatives) will also be retained. As it is widely used for GC, the continued commercial availability is assured (unlike the situation for immobilized 8-hydroxyquinoline). Although the mode of retention is most likely sorbent extraction as DDC is present in excess, Chromosorb does retain metals from slightly alkaline solution in the absence of DDC and so at least one other mechanism of retention is possible. Under the conditions studied, this mechanism appears to be less efficient than the sorbent extraction; however, work in progress [23] has shown that if the retained metal is eluted with acid, enhancements in detection limits of up to two orders of magnitude may be obtained with loading up to flow rates of 12 ml min^{-1}. The material also has possibilities as a support for liquid ion-exchangers [24,25].

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